

CO Oxidation Efficiency and Hysteresis Behavior over Mesoporous Pd/SiO₂ Catalyst

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Supplemental Materials: Influence of Preparation Conditions on CO Conversion Efficiency and Hysteresis behavior of Pd/SiO₂ Aerogel Catalyst

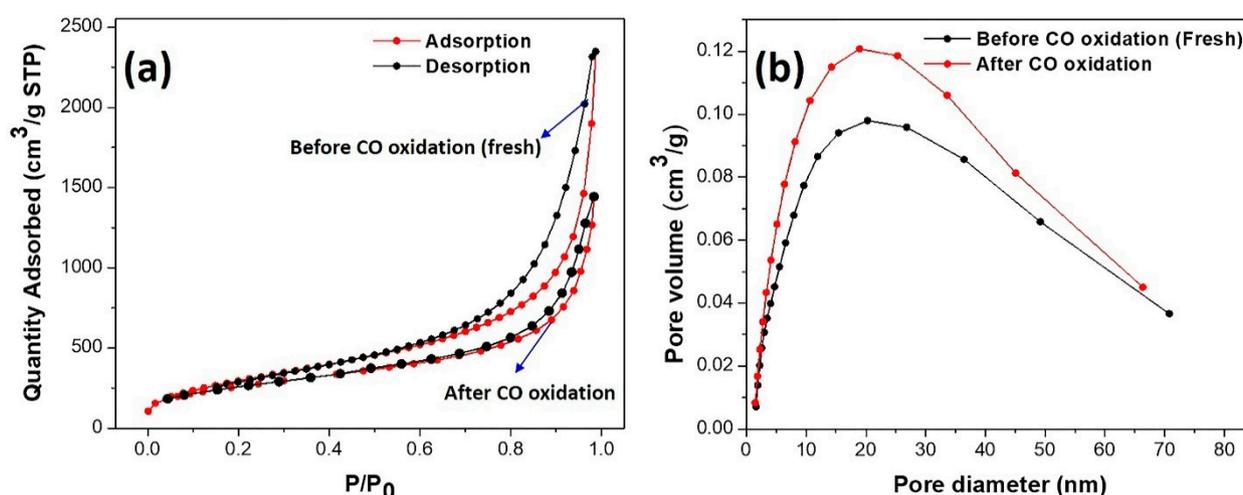


Figure S1. Langmuir Isotherms of N₂ adsorption and desorption (a) and Pore diameter distribution (b) of fresh Pd/SiO₂ aerogel catalyst before and after the catalytic CO oxidation (First light-off).

Figure. S1. Shows that fresh Pd/SiO₂ aerogel catalyst has continuous pore volume distribution with diameters between 2 to 80 nm. However, N₂ adsorption-desorption characteristics of Pd/SiO₂ aerogel pore size distribution after CO oxidation (after first light-off) shows a pore volume with continuous distribution of pore diameters (between 2 -60 nm) that is slightly lower than the distribution for the freshly prepared catalysts. The higher temperature during the CO oxidation does not affect the integrity of the catalyst. However, the quantity of the large macropores and mesopores is eliminated, which collapsed following the heat treatment leading to a larger quantity of micropores and small mesopores as reported by Gage et al. [1]. This was observed in samples as the surface area of the Pd/SiO₂ aerogel reduced to ~940.9 m²/g with an average pore diameter and volume of 15.6 nm and 0.06 cm³/g, respectively. The decrease of the surface area could be due to the sintering of the large excess palladium particles outside the pores.

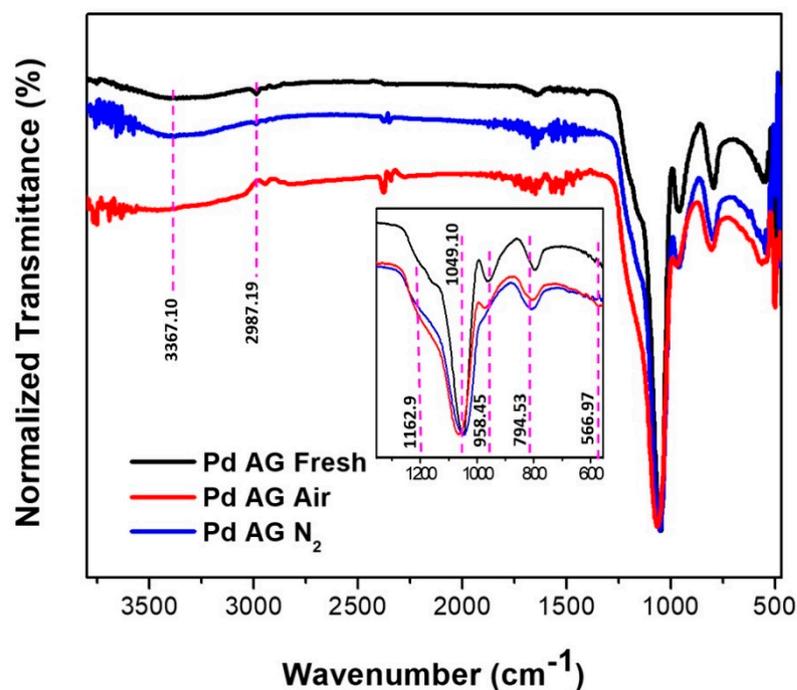


Figure S2. Comparison between FTIR spectra of fresh Pd/SiO₂ aerogel catalysts and after heat treatment at 450 °C in air and nitrogen atmosphere.

Figure S2 compares FTIR spectra of freshly prepared Pd/SiO₂ aerogel catalysts and after heat treatment at 450 °C in air and nitrogen atmospheres. Room-temperature FTIR spectra of Pd/SiO₂ samples measured in spectral range (400–4000 cm⁻¹) were recorded and compared to untreated samples to determine the changes after annealing in N₂ and air atmospheres. The FTIR spectra of both samples revealed several sharp, well-defined absorption bands within the measured spectral range. The spectra show bands centered at 567, 794.5 and 1049.1 cm⁻¹ with a shoulder peak at 1162.9 cm⁻¹ corresponding to stretching vibrations of siloxane groups (Si–O–Si bonds), respectively [2] while the peak centered at 954.5 cm⁻¹ corresponds to the stretching vibration of silanol groups (Si–OH) in the silica lattice suggesting the presence of a considerable amount of silanol groups on the silica surface or pores in all samples. A small peak observed at 2987.2 cm⁻¹ is assigned to the vibrations of the stretching vibration of -CH₃ and -CH₂ groups indicating the presence of a small amount of Si–OC₂H₅ groups can be attributed to an incomplete condensation during gelation [3]. The low-intensity peak at 3367.1 cm⁻¹ is assigned to -OH stretching vibrations [4].

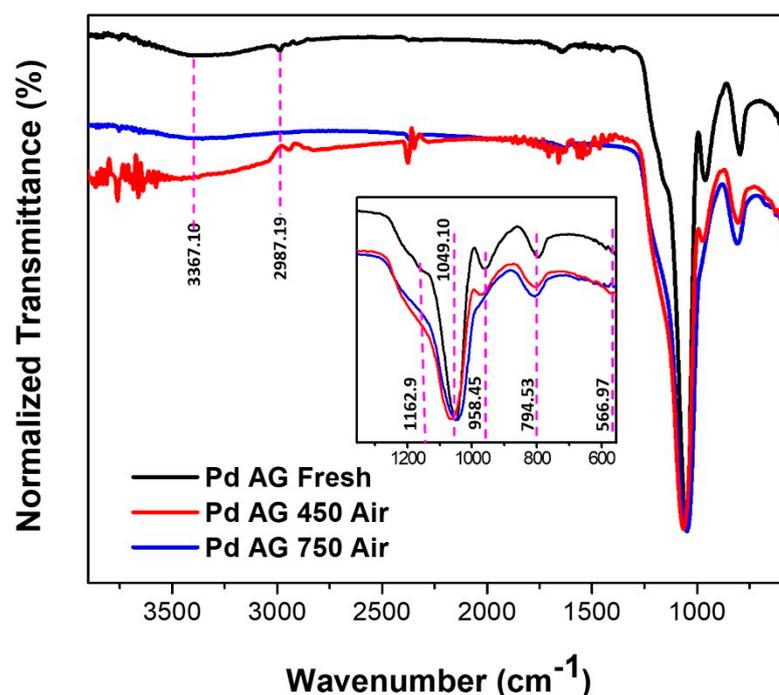


Figure S3. Comparison between FTIR spectra of fresh Pd/SiO₂ aerogel catalysts and after heat treatment at 450 °C and 750 °C in air.

Figure. S3 shows FTIR spectra of freshly prepared Pd/SiO₂ aerogel catalyst and after heat treatment at 450 °C and 750 °C in air, respectively. The infrared absorption is similar to the aforementioned discussion in fig. S2. As the annealing temperature increases, the peak at 958.45 cm⁻¹ slowly disappears, and the intensity of the peak at 2987.2 also decreases, while that of the peak at 794.5 cm⁻¹ increases. This suggests the formation of additional Si–O–Si bonds by the condensation reaction. The peaks at approximately 1710 cm⁻¹ and 3367.1 cm⁻¹ that belong to vibrations of water molecules decrease with increasing annealing temperature due to the removal of water from the SiO₂ network structure [5]. The results suggest that heating to 750 °C could lead to the formation of additional Si–O–Si bonds, which strengthen the SiO₂ network structure [6]. The presence of 958.45 cm⁻¹, which corresponds to the stretching vibration of silanol groups (Si–OH) in the sample heated at 450 °C enhanced the Pd–silica interaction, which could facilitate the dispersion of Pd particles. .

References

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